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NON-CHROMATE SURFACE PREPARATION
OF ALUMINUM

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PREFACE

This report covers the work performed during the period from August 1985 to May 1988 under Air Force Contract No. F33615-84-C-5130. The work was administered under the direction of the Air Force Wright Aeronautical Laboratories, Air Force Materials Laboratory, Wright-Patterson Air Force Base, OH 45433. Messrs. Sam Marolo and Neal Ontko were the Program Engineers. The Principal Investigators on this program were Messrs. Charles Griffen and Robert Askins. The major portion of the laboratory work was conducted by Messrs. Charles Griffen, Gary Andrews and Donald Byrge.



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NON-CHROMATE SURFACE PREPARATION OF ALUMINUM

1. INTRODUCTION

The surface preparation procedure originally developed by Forest Products Laboratories (FPL)^[1], and described in ASTM D2651, Method G, and ASTM E864 involves the etching of aluminum in an aqueous solution of sodium dichromate and sulfuric acid. It has been used for many years to prepare aluminum surfaces for adhesive bonding. The environmental durability of aluminum bonded joints has been improved by supplementing the FPL etch with a phosphoric acid anodizing (PAA) treatment.^[2] This procedure is described in ASTM D3933 and involves the application of an anodizing potential to the metal while it is immersed in a dilute phosphoric acid bath. However, the chromic compounds in the FPL etch solution present disposal problems which have led to a desire for alternative methods of surface preparation. In this program, bonded aluminum samples were prepared with several surface preparation methods which did not include chromic compounds. These bonded samples were tested with the Wedged Crack Extension Test (ASTM D3762), Floating Roller Peel Test (ASTM D3167) and the Double Lap Shear Test (ASTM D3528).

2. BACKGROUND

Many physical and chemical treatments have been used to prepare aluminum surfaces for improved adhesive bonding. The purpose of these treatments is (1) to remove surface contamination in the form of dirt and grease, (2) to increase the macroscopic surface roughness through mechanical abrasion or to increase the microscopic surface roughness through chemical etching, and (3) to improve the resistance of the bonds formed on these surfaces to environmental degradation.

In the case of the phosphoric acid anodization surface treatment, surface contamination is removed in the solvent degreasing and alkaline cleaning steps. The microscopic surface roughness is increased by the deoxidation and the phosphoric acid

anodization steps. The increased environmental resistance is achieved by the nature of the oxide generated on the aluminum surface during anodization and by the prompt application of a primer layer to protect the freshly generated oxide from hydrolytic attack.

An interpretation of the oxide morphology formed by the FPL process on 2024-T3 aluminum is illustrated in Figure 1.^[3] The oxide "whiskers" which promote physical interlocking with the adhesive are evident. Figure 2 illustrates the oxide morphology formed by the PAA treated aluminum surface.^[3] It will be noted that the "whiskers" are much longer and the cells are much deeper, providing an improved mechanical bond.

Upon exposure to moisture, the aluminum oxide progressively hydrates to form boehmite (AlOOH or $\text{Al}_2\text{O}_3 \cdot \text{H}_2\text{O}$), pseudo-boehmite ($\text{Al}_2\text{O}_3 \cdot 2 \text{H}_2\text{O}$), and then bayerite ($\text{Al}(\text{OH})_3$ or $\text{Al}_2\text{O}_3 \cdot 3 \text{H}_2\text{O}$). As hydration proceeds, the adhesion of the oxide to the aluminum decreases and thus gives rise to bond failure. The surface composition of a PAA aluminum adherend consists of a layer of AlPO_4 over the aluminum oxide. The hydration process of the aluminum phosphate is much slower; the first stage involves absorption of water and involves no morphological change and may be reversed by drying in an oven or dessicator. In the second stage, the AlPO_4 layer slowly dissolves and then the aluminum oxide quickly hydrates to pseudo-boehmite. In the third stage, the surface hydrates further to become bayerite. The dissolution of the aluminum phosphate is the rate controlling reaction. While the oxide on an FPL treated aluminum may hydrate in 2 minutes, with the additional PAA treatment, it may take 3 to 5 hours.^[3]

3. TEST PROGRAM

The Wedged-Crack Extension Test was used as a screening test to determine whether a candidate surface treatment looked promising or not. If it did it was repeated to insure

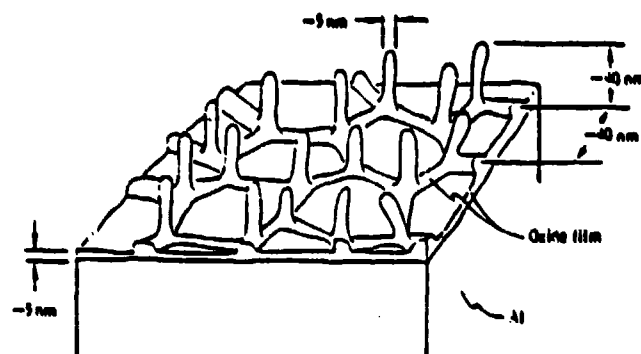


Figure 1. Aluminum Oxide Morphology Formed by Optimized FPL Process [3].

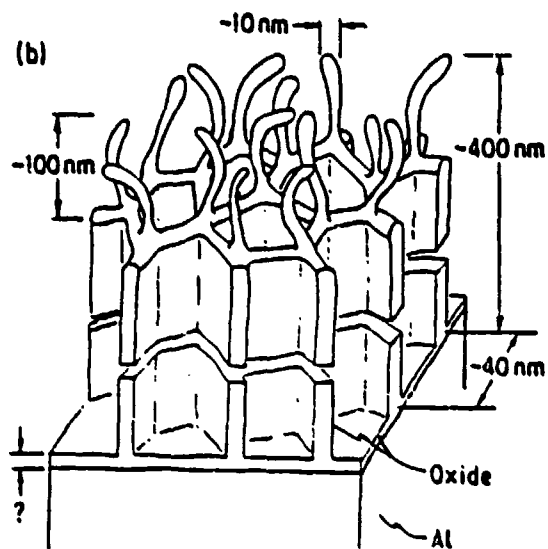


Figure 2. Aluminum Oxide Morphology Formed by PAA Process [3].

reproducibility and then used to prepare peel and lap shear specimens. If not it was dropped from the study.

3.1 Wedge-Crack Extension Test

Altogether, five sets of samples were tested using the standard Wedge Crack Extension Test, ASTM D3762. Three different aluminum alloys were used (2024-T3 Bare, 2024-T3 Clad, and 7075-T6 Bare) with five replicate specimens of each surface preparation. After etching, all of the specimens were primed with BR 127 (American Cyanamid) and bonded with EA9628 adhesive (Dexter Hysol). The adhesive was cured for 60 minutes at 250°F in a hydraulic press using 0.010 inch shims to control the adhesive thickness to a target of 0.008 inch. The test specimen is illustrated in Figure 3. The initial crack tip was marked and its length measured as indicated in Figure 3, using a microscope. The samples were then exposed up to 30 days at 95-100 percent RH and 120°F with periodic measurement of crack extension.

The surface preparations for the five sets of samples are listed below.

Set A

1. Optimized FPL etch (control) (see Appendix I),
2. PAA (control) (see Appendix II),
3. PAA without optimized FPL deoxidizing step,
4. P2 etch (aqueous solution of sulfuric acid and ferric sulfate) (see Appendix III),
5. Alumiprep 33®* (non-flammable phosphoric acid based cleaner, brightener and prepaint conditioner),
6. Concentrated nitric acid (71 percent) etch,
7. Aluminum Jelly®**, and
8. Scotchbrite®*** pad abrasion with MEK wipe.

Set B

1. PAA (control), same as Set A2,
2. PAA without optimized FPL deoxidizing step, same as Set A3,

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** Trademark of Loctite Corp.

*** Trademark of 3M Co.

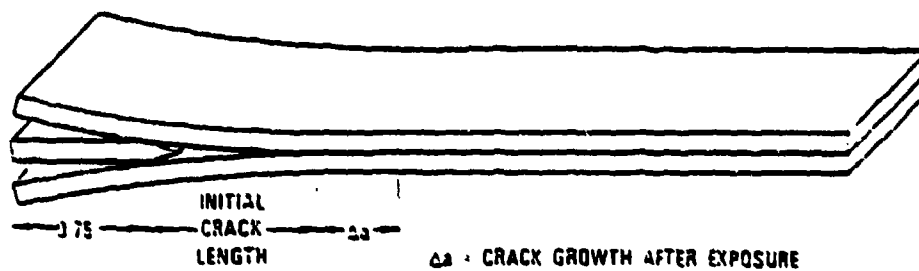


Figure 3. Wedged Crack Extension Specimen.

3. PAA except that sodium dichromate was omitted from the solution used in the deoxidizing step,
4. Dilute nitric acid (10 percent) deoxidizing, and
5. PAA samples which had been anodized and primed for one month prior to the application of the adhesive.

Set C

1. PAA (control), same as Set A2,
2. PAA except that the deoxidizing solution was phosphoric acid rather than the normal sulfuric acid-sodium dichromate solution,
3. PAA except that the deoxidizing step consisted of a phosphoric acid solution in which the current was reversed from the normal anodizing procedure rather than using the normal sulfuric acid-sodium dichromate deoxidizing procedure,
4. PAA except that the sodium dichromate was omitted from the solution used in the deoxidizing step, same as Set B3, and
5. PAA except Scotchbrite® pad abrasion with MEK wipe replaced the optimized FPL etch deoxidizing step.

Set D

1. PAA (control), same as Set A2, and
2. P2 etch, same as Set A4.

Set E (same as Set D)

1. PAA (control), same as Set A2, and
2. P2 etch, same as Set A4.

3.2 Floating Roller Peel Test

After the first three sets of samples were tested with the wedged-crack extension test, two sets of samples were prepared and tested using the standard test method for Floating Roller Peel

Resistance of Adhesives, ASTM D3167. The adherends were 2024-T3 bare aluminum with three replicates for each test condition.

After surface preparation, all of the specimens were primed and bonded as described in Section 3.1. The test specimen is illustrated in Figure 4. The first set was tested at -65°F, 72°F, and 180°F in an unaged condition. The second set was tested at the same conditions and also at -65°F and 180°F after 30 days exposure to 140°F and 95 percent RH. The surface preparations for these two sets of peel samples are described below:

Set D

1. PAA (control), see Set A2 of Section 3.1,
2. Phosphoric acid deoxidizing followed by PAA, see Set C2 of Section 3.1,
3. PAA except that sodium dichromate was omitted from the solution used in the deoxidizing step, see Set B3 of Section 3.1,
4. PAA without optimized FPL deoxidizing step, see Set A3 of Section 3.1, and
5. PAA except Scotchbrite® pad abrasion with MEK wipe replaced the optimized FPL etch deoxidizing step, see Set C5 of Section 3.1.

Set E

Same as Set D except that a sixth group was added using the P2 etch procedure, see Set A4 of Section 3.1.

3.3 Double Lap Shear Tests

Along with the floating roller peel tests, double lap shear tests, ASTM D3528 were run on two sets of samples made with the same surface preparations. The adherends were 2024-T3 bare aluminum with three replicates for each test condition. After surface preparation, all of the specimens were primed and bonded as described in Section 3.1. For the first set, the samples consisted of three pieces of aluminum, 0.063 inch x 1 inch x

4 inch with the adhesive between the overlapping adherends to create two 1/2 inch joints. The second set was similar except that the center adherend was 0.125 inch instead of 0.063 inch (see Figure 5).

The first set was tested at -65°F, 72°F, and 180°F in an unaged condition. The second set was tested at the same conditions and also at -65°F and 180°F after 30 days exposure to 140°F and 95-100 percent relative humidity.

4. RESULTS

4.1 Wedge Crack Extension Tests

The wedge test results are summarized in Table 1 and individual results are given in Appendix IV. In Set A, 19 out of 24 groups had very high crack growth; many of the samples failed during the first hour of exposure. Surface preparation A2, which initially performed poorly, was repeated in sets B and C with excellent results; surface preparation A3 was also repeated in Set B with excellent results. This led to the conclusion that there were some problems in the surface preparation of the samples in Set A.

For the other sets (B, C, D and E), the results are very good for 35 of the 42 groups with an average initial crack length of 1.25 inches (standard deviation of 0.08 inch). The average additional crack length extension during the 30-day high humidity exposure was 0.20 inch. Not included in these averages were the seven groups that had extensive crack growth. These were as follows:

- B2. PAA (no FPL), 7075-T6 Bare
- B4. HNO₃ (10 percent), 2024-T3 Clad
- B4. HNO₃ (10 percent), 7075-T6 Bare
- D2. P2 Etch, 2024-T3 Bare
- D2. P2 Etch, 7075-T6 Bare
- E2. P2 Etch, 2024-T3 Bare
- E2. P2 Etch, 2024-T3 Clad

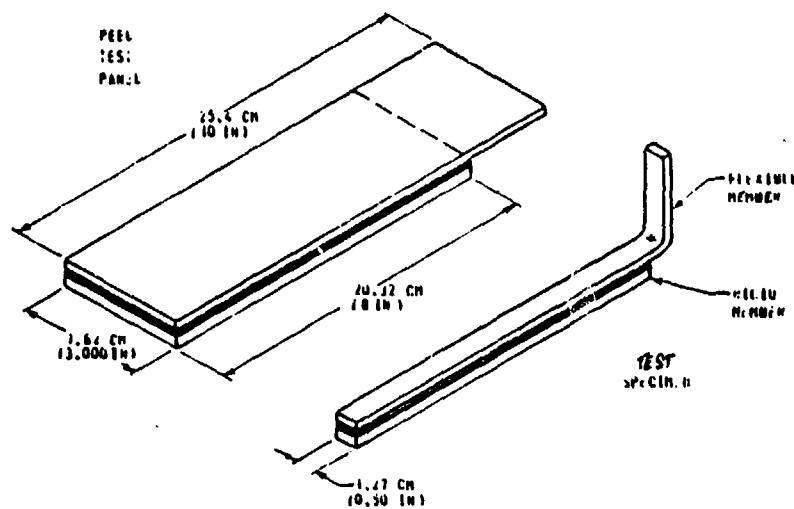


Figure 4. Test Panel and Test Specimen for Floating Roller Peel Test.

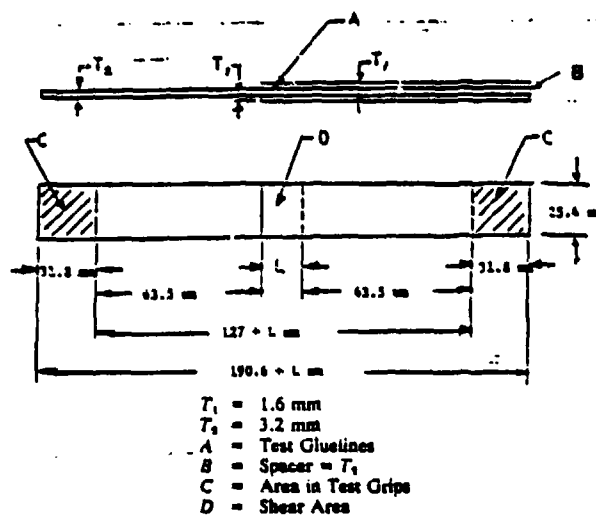


Figure 5. Form and Dimensions of Specimen for Double Lap Shear Test.

SUMMARY RESULTS

NON-CHROMATE SURFACE PREPARATION OF ALUMINUM, WEDGED CRACK EXTENSION TEST

| Surface Preparation | 2024-T3 Bare | | | 2024-T3 Clad | | | 7075-T6 Bare | | |
|--|--------------|---------|-----------------|--------------|--------------------------------|---------|--------------|----------------|-----------------|
| | Init. | 1-4 hrs | 30 days | Init. | 1-4 hrs | 30 days | Init. | 1-4 hrs | 30 days |
| A1. OPT. PPL (Control) | 1.29" | NC | 0.19 | 1.28 | NC | 0.17 | 1.27 | NC | 0.21 |
| A2. PAA (Control) | 1.38" | 2.56 | - | 2.03 | (Fail 1 hr) | - | 1.45 | (Fail 1 hr) | - |
| A3. PAA (No PPL step) | 1.28" | 2.50 | NC | 1.70 | 3.09 {2-0.17 3-Fail 1-4 hrs | - | 1.38 | (Fail 1 hr) | - |
| A4. P2 | 1.31" | 1.73 | NC | 1.31 | NC | 0.13 | 1.29 | NC | NC |
| A5. Alumiprep 33 ① | 1.37" | 2.56 | NC | 1.74 | - | - | 1.53 | - | - |
| A6. Nitric Acid (Conc.) | 1.46" | 2.94 | - | 2.02 | (Fail 1 hr) | - | 1.32 | (Fail 1 hr) | 0.21 |
| A7. Aluminum Jelly ② | 1.25" | 2.49 | NC | 2.05 | {2-2.30 2-Fail 1 hr | 0.20 | 1.45 | 1.97 | NC |
| A8. Scotchbrite ③ | 1.42" | 2.72 | (Fail 1-8 hrs) | 1.48 | {2-2.68 3-Fail 1 hr | NC | 1.37 | 2.49 | NC |
| B1. PAA (Control) | 1.23" | .05 | .15 | 1.23 | .33 | .15 | 1.24 | .34 | .13 |
| B2. PAA (No PPL step) | 1.35" | .04 | .11 | 1.17 | .08 | .18 | 1.62 | - | - |
| B3. PPL w/o Chr. + 2AA | 1.17" | .07 | .15 | 1.16 | .13 | .11 | 1.28 | (Fail 1-4 hrs) | .15 |
| B4. HNO ₃ , 10% | 1.22" | .10 | .84 | 1.24 | 1.54 | .10 | 1.28 | .30 | .50 |
| B5. PAA (Aged 1 Month With Pk13 Before ADH. Applied) | 1.30" | .08 | .14 | 2.09 | - | - | 1.25 | .43 | .11 |
| C1. PAA (Control) ④ | 1.26" | .03 | .01 | 1.27 | .01 | .05 | 1.31 | .01 | .03 |
| C2. P.A. Etch/PAA | 1.28" | .07 | .11 | 1.15 | .02 | .04 | 1.26 | NC | NC |
| C3. REV P.A. Etch/PAA | 1.19" | .06 | .02 | 1.14 | .03 | .02 | 1.30 | .02 | .03 |
| C4. PPL w/o Chr. + PAA | 1.27" | NC | .03 | 1.18 | .05 | NC | 1.24 | NC | .05 |
| C5. Scotchbrite/PAA | 1.26" | .41 | .12 | 1.26 | NC | .05 | 1.29 | .03 | NC |
| D1. PAA (Control) | 1.38" | NC | NC | 1.16 | NC | NC | 1.28 | NC | NC |
| D2. P2 Etch | 2.01" | 1.58 | .23 (2 fail) | 1.06 | .42 | NC | 1.38 | 2.07 | .35 (1 fail) |
| E1. PAA (Control) | 1.47" | NC | .02 | 1.20 | .01 | .01 | 1.35 | .03 | .01 |
| E2. P2 Etch | 1.66" | 1.29 | .05 | 1.23 | 1.55 | .06 | 1.41 | .33 | .42 |

NC = No Change
Results are average of five samples. All samples were primed with BR127 to a thickness of 0.0001-0.0002 inch. All samples were bonded with EA9628 adhesive.
Crack lengths for 1-4 hrs. and 30 days represent additional crack growth.

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② Trademark of Loctite Corp.

③ Trademark of 3M Co.

④ Samples from Set C were exposed an additional six months in salt spray; there were no changes in the crack length during the entire six-month period.

For the initial crack, the failure mode was cohesive on all samples. For the good samples, the failure mode during exposure was cohesive; for the samples with extensive crack growth, the failure mode during exposure was adhesive (interfacial) between the primer and the bare aluminum.

Based on the results for sets B and C, modified versions of the PAA procedure described in ASTM D3933 provided very promising replacement treatments for the non-chromate surface preparation of aluminum for adhesive bonding. These four modified PAA methods are as follows:

1. Phosphoric acid used as deoxidizing solution, followed by PAA.
2. Dilute sulfuric acid deoxidizing followed by PAA.
3. Scuffing the surface with Scotchbrite® pad, followed by MEK solvent wipe and the PAA.
4. Just the PAA treatment (no deoxidizer step).

From these results it appears that phosphoric acid anodization is very tolerant and forgiving of preceding treatment steps. It seems to produce a durable adhesive bond with a wide variety of deoxidizing techniques.

Sets D and E were prepared and tested to double check the inconsistent durability performance of the P2 etch exhibited in Set A (poor on 2024-T3 bare, good on 2024-T3 clad and 7075-T6 bare). Based on the results from sets D and E, the P2 etch did not exhibit satisfactory durability in the wedged-crack extension test.

It is evident from the results of these five sets of samples that the crack growth behavior observed during the first 24 hours of exposure is a very reliable indicator of bond durability over the entire 30-day exposure. This can be utilized in the future as justification for shortening the wedge crack growth time requirements for screening purposes.

It would be very helpful if the various process specifications could be standardized and tighter limits placed on

the concentrations of the materials and times for etching and anodizing. For example, Table 2 lists seven references for the FPL etch and the optimized FPL etch which show the variations in concentrations, especially the sodium dichromate. The first FPL solution is from the original reference and does not contain any aluminum. The only difference between the FPL etch and the optimized FPL etch is the dissolved 2024-T3 aluminum.

TABLE 2
FPL AND OPTIMIZED FPL ETCH SOLUTIONS
GRAM PER LITER

| | <u>Sodium Dichromate</u> | <u>Sulfuric Acid</u> | <u>Aluminum</u> |
|---|------------------------------|--------------------------|-----------------|
| 1. Original FPL etch[1] | 33.7± 3.7 | 337±13.5 | 0 |
| 2. ASTM D2651, Method G | 47.7±19.6 | 299±12.0 | 1.5 |
| 3. ASTM E864-84 | 68.6±41.2 | 274 | 1.6 |
| 4. AFWAL-TR-80-4183 | 31.2 | 274 | 1.9 |
| 5. AFWAL-TR-77-206 | 60.3±29.5 | 300±12.0 | 1.5 |
| 6. FPS-1009D, General Dynamics | 108 | 279 | 0 |
| 7. C2803, Adhesives, Coatings and Sealants Div., 3M Co. | 33.7± 3.0 | 300±12.0 | 0 |

4.2 Floating Roller Peel Test

Based on the results of the wedged-crack extension tests, floating roller peel test samples were prepared using the modified PAA processes. There were several voids in the bondlines of the first set of samples so a second set was prepared. Specimens made with the P2 etch process were also included in the second set. The results for both sets are listed in Table 3. It is evident from the data in Table 3 that the results from the second set are much higher than those from the first. The individual results for the second set are listed in Appendix V, which also includes failure modes.

TABLE 3
SUMMARY RESULTS
NON-CHROMATE SURFACE PREPARATION OF ALUMINUM
FLOATING ROLLER PEEL TEST¹

| Set/ Group No. | Surface Preparation | Peel Strength (lb/in width) | | | | |
|-------------------|-----------------------------------|-----------------------------|------|-------|--------------------|--------------------|
| | | Test Temperature | | | | |
| | | -65°F | 72°F | 180°F | -65°F ² | 180°F ² |
| D1 | PAA (Control) | 38 | 60 | 63 | -- | -- |
| D2 | Phos. Acid Deoxidizing/ PAA | 42 | 59 | 66 | -- | -- |
| D3 | Sulf. Acid Deoxidizing/ PAA | 37 | 63 | 61 | -- | -- |
| D4 | PAA (No Deoxidizing) | 43 | 57 | 58 | -- | -- |
| D5 | Scotchbrite Scour/MEK Wipe/PAA | 40 | 57 | 62 | -- | -- |
| E1 | PAA (Control) | 59 | 73 | 80 | 56 | 80 |
| E2 | Phos. Acid Deoxidizing/ PAA | 55 | 78 | 81 | 56 | 78 |
| E3 | Sulf. Acid Deoxidizing/ PAA | 59 | 83 | 89 | 52 | 83 |
| E4 | PAA (No Deoxidizing) | 59 | 81 | 91 | 55 | 90 |
| E5 | Scotchbrite Scour/MEK Wipe/PAA | 65 | 85 | 95 | 57 | 92 |
| E6 | P2 Etch (No Anodizing) | 52 | 75 | 83 | 51 | 81 |

NOTES:

1. Values represent average of one to three samples. Adherends were 2024T3 bare aluminum. All specimens were primed with BR127 primer to a thickness of 0.0001-0.0002 inch and bonded with EA-9628 adhesive.
2. The additional tests at -65°F and 180°F were run after 30 days exposure to 140°F and 95-100 percent R.H.

For the floating roller peel tests, three aluminum panels were used for each surface preparation. After etching, each of these panels was cut into five samples for a total of 15 samples. For each of the five test conditions, one sample was used from each of the above panels. After testing, it was noted that the peel strengths were low on all 30 samples from six of the 18 panels regardless of the test conditions and surface preparations. It was also noted that the percentage failure mode due to lack of adhesion of metal to primer was high for these samples. It was concluded that the panels did not have a good etch and, therefore, the test results were not included in the summary averages. These samples were E1-PAA(13, 16, 19, 22, 25), E2-PAD/PAA(13, 16, 19, 22, 25), E2-PAD/PAA(14, 17, 20, 23, 26), E4-PAA, no deox.(15, 18, 21, 24, 27), E5-SC, MEK(14, 17, 20, 23, 26) and E5-SC, MEK(15, 18, 21, 24, 27). The individual results in Appendix V are grouped according to the panel from which they were taken rather than by test condition. This was done to illustrate the low peel strength results on the samples from the six panels and the much higher results on the samples from the other 12 panels.

The test results for the samples used in the summary results were similar to the control regardless of the surface preparations. The samples with the Scotchbrite scour/MEK wipe and PAA were consistently slightly higher than the control for this limited sampling. The overall average peel strengths were higher as the test temperature increased and were slightly lower after aging.

The average failure mode for all of the samples used in the summary results were 9 percent metal to primer, 18 percent primer to adhesive, 57 percent cohesive failure of the adhesive, and 16 percent voids. The failure modes at the -65°F test conditions were consistently higher than average on percentage of primer to adhesive failure. At the 180°F test conditions, the failure modes were much higher on cohesive failure of the adhesive and lower on lack of adhesion of metal to primer.

The failure modes for the samples with sulfuric acid etch and with the Scotchguard scour were similar to those of the control. For the samples with the phosphoric acid etch, the failures were higher than average on the percentage of adhesion of the metal to primer and lower than average on the percentage of adhesion of the primer to the adhesive. The samples with the phosphoric acid anodization but with no deoxidation step, had a higher percentage of cohesive failure of the adhesive and lower than average percentage failure of adhesion of the primer to the adhesive. The samples with the P2 etch had higher than average percentage failure of metal to primer and of primer to adhesive but lower than average percentage of cohesive failure of the adhesive.

4.3 Double Lap Shear Test

The same surface preparation processes were used for preparing double lap shear specimens as for the floating roller peel specimens. The summary results for these tests are listed in Table 4. The individual results and failure modes are tabulated in Appendix VI.

The results for the samples with the sulfuric acid and with no deoxidizing step prior to PAA (E3 and E4) were slightly lower than the results for the other four preparations. The rest of the results were generally within 10 percent of each other for all of the test conditions. Failure modes were 85-90 percent cohesive for all cases. This high level of cohesive failure indicates that the slight differences observed in strength were probably not due to differences in surface preparation but to other factors.

5. CONCLUSIONS AND RECOMMENDATIONS

The P2 etched samples were equivalent in peel and lap shear to the standard phosphoric acid anodized surfaces but they did not exhibit satisfactory durability in the wedged crack extension tests. Of all the other non-chromate surface preparations tested,

TABLE 4
SUMMARY RESULTS
NON-CHROMATE SURFACE PREPARATION OF ALUMINUM
DOUBLE LAP SHEAR TEST RESULTS¹

| Set/ Group No. | Surface Preparation | Lap Shear Strength (psi) | | | |
|-------------------|------------------------------------|--------------------------|------|-------|-------------------------|
| | | Test Temperature | | | |
| | | -65°F | R.T. | 180°F | 180°F, wet ² |
| E1 | PAA (Control) | 7870 | 7120 | 6070 | 4190 |
| E2 | Phosphoric Acid Etch/PAA | 7940 | 6890 | 5910 | 3800 |
| E3 | Sulfuric Acid Etch/PAA | 6980 | 5950 | 4950 | 3770 |
| E4 | PAA (No Deoxidizing) | 7390 | 5750 | 4960 | 2820 |
| E5 | Scotchbrite Scour/ MEK Wipe/PAA | 8300 | 6890 | 6080 | 4690 |
| E6 | P2 Etch (No Anodizing) | 7070 | 5550 | 5270 | 3260 |

NOTES:

1. Results represent an average of 3 specimens. Adherends were 2024-T3 bare (1/8 inch center adherend, 1/16 inch outside adherends). All samples were primed with BR127 primer to a thickness of 0.0001-0.0002 inch and bonded with EA-9628 adhesive.
2. Tested at 180°F after 30 days exposure to 140°F and 95-100 percent R.H.

the four best were some non-chromate containing modifications of the standard phosphoric acid anodization (PAA) process. These four modified procedures differed from the standard PAA process only in that the deoxidation step was changed. Three of these four modified procedures appeared equivalent to the standard PAA process, while the fourth was only slightly inferior. The three best involved the substitution of phosphoric acid, sulfuric acid, or a mechanical surface abrasion for the normal sulfuric acid/sodium dichromate deoxidation treatment. The fourth, and slightly inferior approach, simply omitted the deoxidation step completely. The necessity of including the deoxidizing step should probably be further pursued since Venables [3] noted that "an FPL oxide dissolves completely within 30 seconds after immersion in the PAA electrolyte".

REFERENCES

1. H.W. Eichner and W.E. Schowalter, Forest Products Laboratory, Madison, WI, Report No. 1813 (1950).
2. G.S. Kabayashi and D.J. Donnelly, Boeing Co., Seattle, WA, Report No. DG-41517 (February 1974).
3. J.D. Venables, J. Materials Science, 19(1984), 2438.
4. J.D. Venables, et al., Appl. Surf. Sci., 3(1979), 88.

APPENDIX I

OPTIMIZED FPL ETCH

The optimized Forest Products Laboratory (FPL) procedure as listed in ASTM D2651, Method G and ASTM E864 are given below. For further details, consult the complete method in the ASTM manuals. This procedure is used to prepare aluminum surfaces for adhesive bonding.

ASTM D2651, METHOD G

5.7 Method G - A successful and widely used method of surface preparation for structural adhesive bonding of 2024, 7075, and 6061 aluminum alloys is as follows:

5.7.1 Vapor degrease, emulsion degrease, or solvent wipe as required by the conditions of the metal (Caution, see 5.7.3.2). This step may be omitted when the metal is not obviously oily or ink marked.

5.7.2 Immerse in a nonsilicated, nonetching, free-rinsing alkaline cleaner in accordance with the cleaner manufacturer's recommendation. Spray or immersion rinse thoroughly without delay with demineralized water (minimum 70°F). (Cleaner must not dry on surface prior to rinsing.) Parts shall exhibit a water break-free surface.

5.7.3 Immerse in sodium dichromate/sulfuric acid etch solution (Caution, see 5.7.3.2) for 12 to 15 minutes at (150 to 160°F, 66 to 71°C). Do not allow details to dry between etch and rinse cycles.

5.7.3.1 Sodium Dichromate/Sulfuric Acid Etch:

| <u>Material</u> | <u>Concentration</u> |
|--------------------------|--|
| Sulfuric acid 66°Be | 38.5-41.5 oz/gal (287.9-310.0 g/litre) |
| Sodium dichromate | 4.1-9.0 oz/gal (28-67.3 g/litre) |
| Aluminum Alloy-2024 bare | 0.2 oz/gal (1.5 g/litre) minimum (150-160°F) (66-71°C) |

5.7.3.2 Caution - This is a hazardous material; exercise proper precaution when handling.

5.7.4 Rinse thoroughly in demineralized water at 70°F (21°C) to 130°F (54°C).

5.7.5 Allow parts to dry thoroughly at temperatures up to 140°F (60°C) maximum.

5.7.6 Handle all detail parts that have been processed for adhesive bonding with clean, white cotton gloves only.

ASTM E864

5.2 Etch Solution:

5.2.1 Method I, Sulfo-Chromate Etch (FPL) - The chemical analysis of the etch solution shall be maintained at approximately 30 parts by mass of water, 10 parts by mass of sulfuric acid (sp gr 1.84), and one to four parts by mass of sodium dichromate. Prior to use, a minimum of 0.06 part by mass of dissolved 2024 aluminum shall be added.

7.1.1 Degreasing - Remove any visible oil and grease from the aluminum by vapor degreasing or solvent cleaning. Vapor degrease using a degreasing solvent. Remove the parts being degreased from the degreaser as soon as condensation ceases. Solvent clean using safety solvent (Fed. Std. O-T-620) at room temperature.

7.1.2 Alkaline Cleaning - Immerse the aluminum in the alkaline cleaning solution held from 50 to 80°C (122 to 176°F) for a minimum of 5 minutes. Follow the alkaline cleaning by thorough rinsing in water from room temperature to 70°C (158°F). Alkaline cleaning may be repeated. Keep the parts wet between the alkaline treatment and immersion in the rinse tank.

7.1.3 Etching:

7.1.3.1 Sulfo-Chromate Etch - Immerse the aluminum parts in the etch solution for 9 to 15 minutes from 65 to 70°C (149 to 158°F). Keep the parts wet between the etch tank and the rinse tank. Follow the etching by thorough rinsing with room temperature water.

7.1.3.3 Rework - Parts that are rerun, because of lack of water break, stains, or unprimed parts that have exceeded the permitted storage time, may be reworked no more than two times. Do not exceed a total immersion time of 34 minutes.

7.1.4 Final Rinse - Rinse the aluminum for 1 to 2 minutes in water from room temperature to 50°C (122°F).

NOTE 2 - Check parts that can be readily observed for water break and recycle if a water break occurs.

7.1.5 Drying - Air dry the aluminum for not more than 1 hour at a temperature not exceeding 65°C (149°F) prior to movement to the controlled area.

7.2 Restrictions:

7.2.1 Immerse parts completely in all solutions.

7.2.2 Do not allow rinsing stains and dichromate stains on the bonding surfaces.

7.2.3 Place cleaned parts within 1/2 hour in a controlled area with filtered air that is maintained at a relative humidity of 50 percent maximum. Prime or bond the cleaned parts within 16 hours.

Procedure for Surface Preparation of Aluminum Alloys Using the Sulfuric Acid-Sodium Dichromate Etch

STEP 1 - DEGREASE (see 7.1.1)

Vapor degrease or clean with safety solvent. Flush all hollow sections. Repeat as necessary.

STEP 2 - ALKALINE CLEAN (see 7.1.2)

Immerse in aluminum cleaner from 50 to 80°C (122 to 176°F) for 5 to 10 minutes. Repeat as necessary.

STEP 3 - RINSE (see 7.1.3)

Use water from room temperature to 70°C (158°F).

STEP 4 - ETCH (see 7.1.3)

Immerse in sulfuric acid-sodium dichromate etch for 9 to 15 minutes from 65 to 70°C (149 to 158°F). Spray rinse heavy sections as they emerge from the solution as necessary to prevent staining.

STEP 5 - RINSE (see 7.1.3)

Use room temperature water.

STEP 6 - FINAL RINSE (see 7.1.4)

Use water from room temperature to 50°C (122°F) for 1 to 2 minutes.

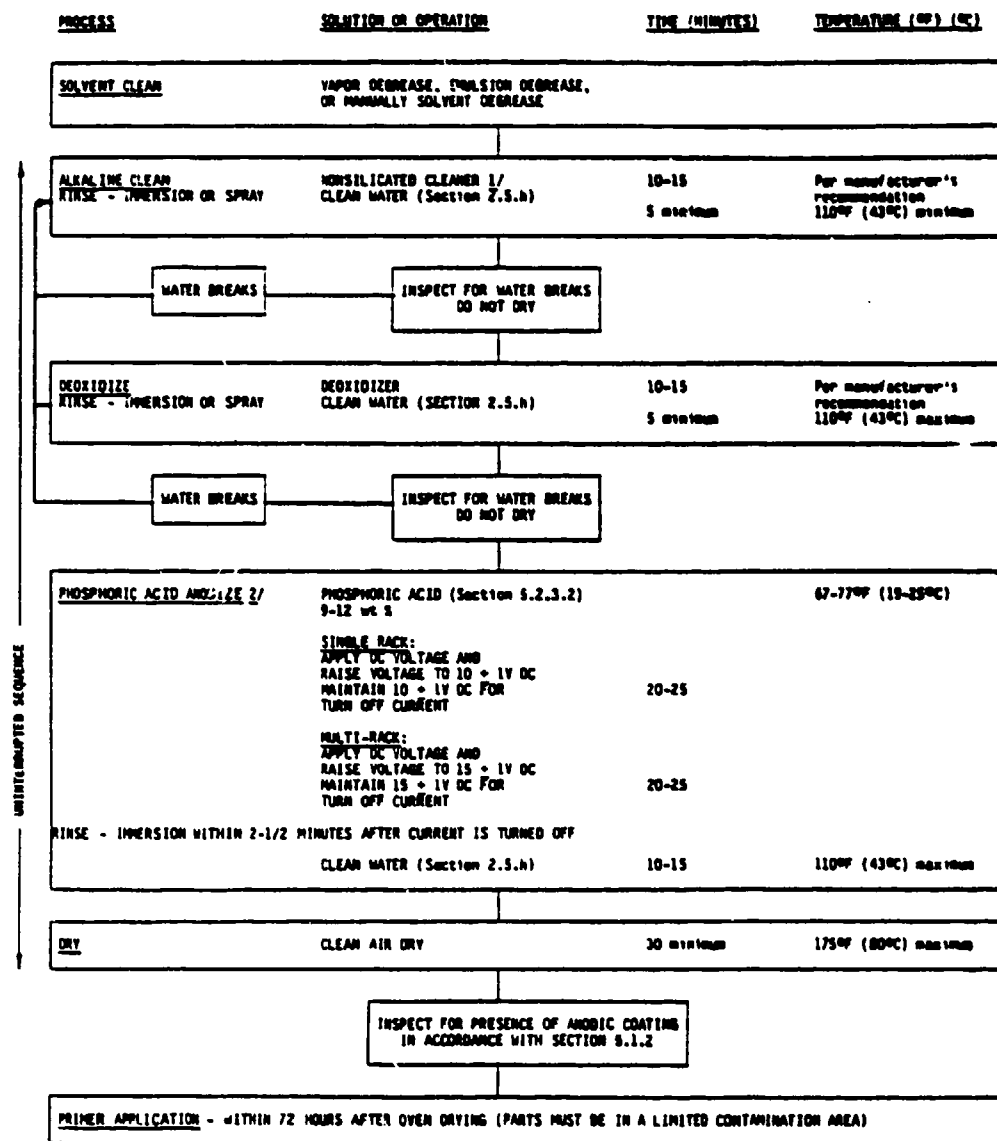
STEP 7 - DRY (see 7.1.5)

Use air from room temperature to 65°C (149°F) for not more than 1 hour.

APPENDIX II

PHOSPHORIC ACID ANODIZATION (PAA)

The phosphoric acid anodizing procedure, as listed in ASTM D3933, is given below. For further details, consult the complete method as listed in the ASTM manual. This procedure is used to prepare aluminum surfaces for adhesive bonding and often uses the optimized FPL etch (Appendix I) as the deoxidizer.



NOTE 1—Experience has proved nonsilicated cleaners to be preferable when reprocessing might be involved.
 NOTE 2—A single-rack facility is one with a single anode rack between each set of cathodes. A multi-rack has two or three anode racks between each set of cathodes.

APPENDIX III

P2 ETCH

The P2 etch procedure as listed in ASTM E864-84 is given below. For further details, consult the complete method in the ASTM manual. This procedure is used to prepare aluminum surfaces for adhesive bonding as an alternate to the optimized FPL etch; this solution contains no chromates.

5.2.2 Method II, Sulfo-Ferric Etch (P_2) - The chemical analysis of the etch solution shall be maintained at approximately 27 to 36 percent by weight (sp. gr. 1.84) of sulfuric acid and 100 ± 25 g/L (3.5 ± 0.9 oz/qt) of ferric sulfate.

7.1.1 Degreasing - Remove any visible oil and grease from the aluminum by vapor degreasing or solvent cleaning. Vapor degrease using a degreasing solvent. Remove the parts being degreased from the degreaser as soon as condensation ceases. Solvent clean using safety solvent (Fed. Std. O-T-620) at room temperature.

7.1.2 Alkaline Cleaning - Immerse the aluminum in the alkaline cleaning solution held from 50 to 80°C (122 to 176°F) for a minimum of 5 minutes. Follow the alkaline cleaning by thorough rinsing in water from room temperature to 70°C (158°F). Alkaline cleaning may be repeated. Keep the parts wet between the alkaline treatment and immersion in the rinse tank.

7.1.3 Etching:

7.1.3.2 Sulfo-Ferric Etch - Immerse the aluminum parts in the etch solution for 10 to 12 minutes from 60 to 65°C (140 to 149°F). Keep the parts wet between the etch tank and the rinse tank. Follow the etching by thorough rinsing with room temperature water.

7.1.3.3 Rework - Parts that are rerun, because of lack of water break, stains, or unprimed parts that have exceeded the permitted storage time, may be reworked no more than two times. Do not exceed a total immersion time of 34 minutes.

7.1.4 Final Rinse - Rinse the aluminum for 1 to 2 minutes in water from room temperature to 50°C (122°F).

NOTE 2 - Check parts that can be readily observed for water break and recycle if a water break occurs.

7.1.5 Drying - Air dry the aluminum for not more than 1 hour at a temperature not exceeding 65°C (149°F) prior to movement to the controlled area.

7.2 Restrictions:

7.2.1 Immerse parts completely in all solutions.

7.2.2 Do not allow rinsing stains and dichromate stains on the bonding surfaces.

7.2.3 Place cleaned parts within 1/2 hour in a controlled area with filtered air that is maintained at a relative humidity of 50 percent maximum. Prime or bond the cleaned parts within 16 hours.

Procedure for Surface Preparation of Aluminum Alloys Using the Sulfo-Ferric Etch

STEP 1 - DEGREASE (see 7.1.1)

Vapor degrease or clean with safety solvent. Flush all hollow sections. Repeat as necessary.

STEP 2 - ALKALINE CLEAN (see 7.1.2)

Immerse in aluminum cleaner at 50 to 80°C (122 to 176°F) for 5 to 10 minutes. Repeat as necessary.

STEP 3 - RINSE (see 7.1.2)

Use water from room temperature to 70°C (158°F).

STEP 4 - ETCH (see 7.1.3.2)

Immerse in the sulfo-ferric etch for 10-12 minutes from 60 to 65°C (140 to 149°F). Spray rinse heavy sections as they emerge from the solution as necessary to prevent staining.

STEP 5 - RINSE (see 7.1.3.2)

Use room temperature water.

STEP 6 - FINAL RINSE (see 7.1.4)

Use water from room temperature to 50°C (122°F) for 1 to 2 minutes.

STEP 7 - DRY (see 7.1.5)

Use air from room temperature to 65°C (149°F) for not more than 1 hour.

APPENDIX IV

Listed in Appendix IV are the individual results
for the wedged crack extension test for
Sets A, B, C, D and E.

APPENDIX IV
INDIVIDUAL WEDGED CRACK EXTENSION TEST RESULTS FOR SET A

I. 2024 T3 Bare Aluminum

| Surf. Prep. | Init. | Wedged Crack Extension, inches | | | | | | | | | |
|---------------------------|-------|--------------------------------|-------|-------|--------|--------|--------|-------|--------|--------|---------|
| | | 1 hr. | 4 hr. | 8 hr. | 24 hr. | 48 hr. | 72 hr. | 1 wk. | 2 wks. | 3 wks. | 30 days |
| 1. OFPL (control) | 1.33 | - | - | - | - | - | - | - | .12 | - | - |
| | 1.22 | - | - | - | - | - | - | - | .29 | - | - |
| | 1.28 | - | - | - | - | - | .16 | .23 | - | - | - |
| | 1.34 | - | - | - | - | .15 | - | - | - | - | - |
| | 1.29 | - | - | - | - | .03 | .03 | .05 | .08 | - | - |
| 2. PAA (control) | 1.39 | 3.70 | Fail | - | .60 | .10 | - | - | - | - | - |
| | 1.35 | 2.21 | .27 | - | - | - | - | - | - | - | - |
| | 1.37 | Fail | - | - | - | - | - | - | - | - | - |
| | 1.41 | Fail | - | - | - | - | - | - | - | - | - |
| | 1.38 | 1.77 | Fail | - | .60 | .10 | - | - | - | - | - |
| 3. PAA w/o OFPL deox. | 1.25 | 3.81 | - | Fail | - | - | - | - | - | - | - |
| | 1.30 | .70 | 1.81 | - | - | - | - | - | - | - | - |
| | 1.28 | 2.45 | - | - | - | - | - | - | - | - | - |
| | 1.23 | 3.19 | - | - | - | - | - | - | - | - | - |
| | 1.35 | .39 | 2.34 | - | - | - | - | - | - | - | - |
| 4. P2 | 1.28 | 4.11 | 0.83 | - | - | - | - | - | - | - | - |
| | 1.34 | .16 | 1.81 | - | - | - | - | - | - | - | - |
| | 1.31 | 1.11 | - | - | - | - | - | .21 | - | - | - |
| | 1.25 | 1.78 | - | - | - | - | - | - | .11 | - | - |
| | 1.35 | 1.92 | - | - | - | - | - | - | - | - | - |
| 5. Alumi- prep 33 | 1.31 | .15 | 1.70 | - | - | - | - | .04 | .02 | - | - |
| | 1.31 | 1.02 | 0.70 | - | - | - | - | - | - | - | - |
| | 1.65 | 2.86 | - | .10 | - | - | - | - | - | .12 | - |
| | 1.36 | .57 | 1.06 | - | - | .12 | - | - | - | - | - |
| | 1.30 | 2.75 | - | - | - | - | - | - | - | .13 | - |
| 6. HNO ₃ (71%) | 1.28 | 1.98 | .55 | - | - | - | - | - | - | - | - |
| | 1.27 | 2.42 | .59 | - | - | .02 | - | - | - | .05 | - |
| | 1.37 | 2.12 | .44 | .02 | - | - | - | - | - | - | - |
| | 1.55 | 3.45 | - | Fail | - | .12 | - | - | - | - | - |
| | 1.38 | 2.42 | .45 | - | - | - | - | - | - | - | - |
| | 1.42 | Fail | - | - | - | - | - | - | - | - | - |
| | 1.45 | Fail | - | - | - | - | - | - | - | - | - |
| | 1.51 | 2.96 | Fail | - | - | .12 | - | - | - | - | - |
| | 1.46 | 2.94 | .23 | - | - | - | - | - | - | - | - |

APPENDIX IV (Continued)

| Surf. Prep. | Init. | 1 hr. | 4 hr. | 8 hr. | 24 hr. | 48 hr. | 72 hr. | 1 wk. | 2 wks. | 3 wks. | 30 days |
|-------------------------------|-------|-------|-------|-------|--------|--------|--------|-------|--------|--------|---------|
| Wedge Crack Extension, Inches | | | | | | | | | | | |
| 7. Aluminum Jelly | 1.23 | 1.95 | - | - | - | - | - | - | - | - | - |
| | 1.26 | 1.52 | - | .68 | - | - | - | - | - | - | - |
| | 1.26 | 2.98 | - | - | - | - | .20 | - | - | - | - |
| | 1.28 | 2.87 | - | - | - | - | - | - | - | - | - |
| | 1.22 | 3.14 | - | - | - | - | - | - | .16 | - | - |
| | 1.25 | 2.49 | - | .14 | - | - | .04 | - | .03 | - | - |
| 8. Scotch-brite, MEK Wipe | 1.41 | 3.06 | - | - | - | - | - | - | - | - | - |
| | 1.39 | Fail | - | - | - | - | - | - | - | - | - |
| | 1.38 | 2.64 | - | Fail | - | - | - | - | - | - | - |
| | 1.53 | 2.45 | - | Fail | - | - | - | - | - | - | - |
| | 1.41 | Fail | - | - | - | - | - | - | - | - | - |
| | 1.42 | 2.72 | - | - | - | - | - | - | - | - | - |
| II. 2024 T3 Clad Aluminum | | | | | | | | | | | |
| 1. OPPL (control) | 1.35 | - | - | - | - | - | .13 | - | - | - | - |
| | 1.32 | - | - | - | - | - | .33 | - | - | - | - |
| | 1.23 | - | - | - | - | - | - | .22 | - | - | - |
| | 1.23 | - | - | - | - | .18 | - | - | - | - | - |
| | 1.25 | - | - | - | - | .04 | .09 | .04 | - | - | - |
| | 1.28 | - | - | - | - | - | - | - | - | - | - |
| 2. PAA (control) | 2.08 | Fail | - | - | - | - | - | - | - | - | - |
| | 1.83 | Fail | - | - | - | - | - | - | - | - | - |
| | 2.09 | Fail | - | - | - | - | - | - | - | - | - |
| | 2.21 | Fail | - | - | - | - | - | - | - | - | - |
| | 1.92 | Fail | Fail | - | - | - | - | - | - | - | - |
| | 2.03 | 1.26 | - | - | - | - | - | - | - | - | - |
| 3. PAA w/o OPPL deox. | 1.20 | 3.10 | - | - | .21 | - | - | - | - | - | - |
| | 1.80 | 1.01 | 1.81 | - | - | - | - | - | - | - | - |
| | 1.85 | Fail | - | - | - | - | - | - | - | - | - |
| | 1.80 | 2.81 | Fail | - | - | - | - | - | - | - | - |
| | 1.83 | 2.18 | Fail | - | - | - | - | - | - | - | - |
| | 1.70 | 2.53 | 0.90 | - | .10 | - | - | - | - | - | - |
| 4. P2 | 1.31 | - | - | - | - | - | - | - | - | - | .18 |
| | 1.26 | .12 | - | - | - | - | - | .16 | - | - | - |
| | 1.35 | - | - | - | - | - | .23 | - | - | - | - |
| | 1.28 | - | - | - | - | .14 | - | - | - | - | - |
| | 1.31 | - | - | - | - | .03 | .05 | .03 | - | - | .04 |
| | 1.31 | 1.02 | - | - | - | - | - | - | - | - | - |

APPENDIX IV (Continued)

| Surf. Prep. | Init. | 1 hr. | 4 hr. | 8 hr. | 24 hr. | 48 hr. | 72 hr. | 1 wk. | 2 wks. | 3 wks. | 30 days |
|----------------------------------|-------|-------|-------|-------|--------|--------|--------|-------|--------|--------|---------|
| Wedge Crack Extension, Inches | | | | | | | | | | | |
| 5. Alumi- prep 33 | 1.75 | 3.42 | - | Fail | - | - | - | - | - | - | - |
| | 2.18 | Fail | - | - | - | - | - | - | - | - | - |
| | 1.45 | Fail | - | - | - | - | - | - | - | - | - |
| | 1.39 | Fail | - | - | - | - | - | - | - | - | - |
| | 1.91 | Fail | - | - | - | - | - | - | - | - | - |
| 6. HNO ₃ (71%) | 1.74 | 3.42 | - | - | - | - | - | - | - | - | - |
| | 2.05 | 2.47 | - | .15 | - | - | - | - | - | - | - |
| | 1.92 | 1.89 | .29 | - | - | 0.00 | - | - | - | - | - |
| | 2.12 | Fail | - | - | - | - | - | - | - | - | - |
| | 1.92 | 2.27 | - | - | - | - | - | - | - | - | - |
| 7. Aluminum Jelly | 2.08 | Fail | .10 | .05 | - | 0.03 | - | - | - | - | - |
| | 2.02 | 2.21 | - | - | - | - | - | - | - | - | - |
| | 1.68 | 3.37 | - | .09 | Fail | - | - | - | - | - | - |
| | 1.52 | Fail | - | - | - | - | - | - | - | - | - |
| | 1.58 | Fail | - | - | - | - | - | - | - | - | - |
| 8. Scotch- brite, MEK Wipe | 1.48 | Fail | Fail | - | - | - | - | - | - | - | - |
| | 1.47 | 1.87 | - | - | - | - | - | - | - | - | - |
| | 1.53 | 2.62 | - | .09 | - | - | - | - | - | - | - |
| | 1.70 | 2.59 | - | - | - | - | - | - | - | - | - |
| | 1.36 | Fail | - | - | - | - | - | - | - | - | - |
| III. 7075 T6 Bare Aluminum | | | | | | | | | | | |
| 1. OFPL (control) | 1.35 | - | - | - | - | - | - | - | - | .22 | - |
| | 1.16 | .07 | - | .09 | - | - | - | - | - | - | - |
| | 1.18 | - | - | - | - | - | - | .30 | - | - | - |
| | 1.29 | - | - | - | - | - | - | .17 | - | - | - |
| | 1.35 | - | - | - | - | - | - | - | .19 | - | - |
| 2. PAA (control) | 1.27 | .01 | - | .02 | - | - | - | .09 | .03 | .04 | - |
| | 1.67 | 3.40 | - | Fail | - | - | - | - | - | - | - |
| | 1.36 | Fail | - | - | - | - | - | - | - | - | - |
| | 1.39 | Fail | - | - | - | - | - | - | - | - | - |
| | 1.46 | Fail | - | - | - | - | - | - | - | - | - |
| | 1.36 | Fail | - | - | - | - | - | - | - | - | - |
| | 1.45 | 3.40 | - | - | - | - | - | - | - | - | - |

APPENDIX IV (Continued)

| Surf. Rep. | Init. | 1 hr. | 4 hr. | 8 hr. | hr. | 48 hr. | 72 hr. | 1 wk. | 2 wks. | 3 wks. | 30 days |
|----------------------------------|--|--|------------------------------------|---------------------------------|---------------------------------|---|----------------------------|----------------------------|---------------------------------|----------------------------------|----------------------------|
| Wedge Crack Extension, Inches | | | | | | | | | | | |
| 3. PAA w/o OFPL deox. | 1.45 1.29 1.38 1.37 1.40 1.35 1.38 | 3.73 Fail Fail Fail 1.35 2.54 | - 1.22 .61 | - - - | Fail | - 0.10 .10 | - - - | - - - | - - - | - - - | - - - |
| 4. P2 | 1.40 1.24 1.31 1.28 1.22 1.29 | - - - - - - | - - - - - - | - - - - - - | - - - - - - | - - - - - - | - - - - - - | - - - - - - | .21 - - - - - | - - - - - - | - - - - - - |
| 5. Alumi- prep 33 | 1.53 1.46 1.63 1.46 1.56 1.53 | Fail Fail Fail Fail Fail Fail | - - - - - - | - - - - - - | - - - - - - | - - - - - - | - - - - - - | - - - - - - | - - - - - - | - - - - - - | - - - - - - |
| 6. HNO ₃ (71%) | 1.37 1.37 1.30 1.24 1.33 1.32 | 2.26 0.79 2.66 2.49 0.73 1.39 | - .71 - - 1.11 0.36 | .10 - - - - -.02 | - .36 - - - -.07 | - .15 - - - -.23 -.08 | - - - - - - | - - - - - - | - - - - - - | - - - - -.23 -.05 | - - - - - - |
| 7. Aluminum Jelly | 1.58 1.35 1.43 1.50 1.39 1.45 | 2.28 2.14 1.63 2.05 1.07 1.83 | - .66 - - -.70 -.27 | - - - - - - | - - - - - - | - - - - - - | - - - - - - | - - - - - - | .23 - - - - -.03 | - - - - - - | - - - - - - |
| 8. Scotch- brite, MEK Wipe | 1.43 1.32 1.37 1.42 1.33 1.37 | 2.76 1.80 2.90 Fail Fail 2.49 | - .73 - - -.24 | - - - - - | Fail .57 - - -.19 | - - - - - | - - - - - | - - - - - | - - - - - | - - -.21 - -.11 | - - - - - |

- = No Change
OFPL = Optimized Forest Products Laboratory Method
PAA = Phosphoric Acid Anodize
P2 = Sulfuric Acid, Ferric Sulfate Etch

APPENDIX IV (Continued)
INDIVIDUAL WEDGED CRACK EXTENSION TEST RESULTS FOR SE. B

I. 2024T3 Bare Aluminum

| Surf. Prep. | Init. | 1 hr. | 4 hr. | 8 hr. | 1 day | 2 days | 3 days | 7 days | 14 days | 21 days | 30 days |
|--|--------|-------|-------|--------|-------|--------|--------|--------|---------|---------|---------|
| Wedged Crack Extension, inches | | | | | | | | | | | |
| 1. PAA (control) | 1.1990 | .0370 | - | - | .0625 | .0495 | - | - | - | - | .0881 |
| | 1.1981 | .0360 | - | .0606 | - | - | - | .0474 | - | - | .1003 |
| | 1.2871 | .0415 | - | - | .0362 | - | - | - | .0403 | - | .0873 |
| | 1.2458 | .0364 | - | - | - | - | - | - | - | - | .0622 |
| | 1.2172 | .0587 | .0430 | - | - | - | - | .0543 | - | - | .0619 |
| | 1.2294 | .0423 | .0086 | .0121 | .0197 | .0099 | - | .0203 | .0081 | - | .0800 |
| 2. PAA w/o OFPL deox. | 1.2878 | .0614 | - | - | - | .0450 | - | - | - | .1481 | - |
| | 1.3539 | - | .0430 | - | - | .0529 | - | - | .0493 | - | .0323 |
| | 1.3049 | .0396 | - | - | .0370 | .0640 | - | - | - | - | .0378 |
| | 1.3446 | .0178 | - | - | .0245 | - | .0504 | - | - | - | .1075 |
| | 1.4800 | .0428 | - | - | - | .0567 | - | - | - | - | .1375 |
| | 1.3542 | .0323 | .0086 | 0 | .0123 | .0437 | .0101 | - | .0099 | .0296 | .0630 |
| 3. H ₂ SO ₄ deoxid. | 1.1454 | .0180 | .0260 | .0214 | .0483 | - | - | .0295 | - | - | .0542 |
| | 1.1336 | - | .0607 | .0527 | - | .0358 | - | - | - | - | .0158 |
| | 1.1189 | .0647 | .0388 | .0368 | - | .0512 | - | - | .0416 | - | .0764 |
| | 1.2259 | .0406 | .0162 | .0391 | - | - | - | - | - | .1158 | .0337 |
| | 1.2039 | .0925 | - | - | - | - | - | .0513 | - | - | .0657 |
| | 1.1655 | .0432 | .0283 | .0300 | .0097 | .0174 | - | .0162 | .0083 | .0232 | .0492 |
| 4. HNO ₃ (10%) | 1.1529 | .1393 | - | 1.3610 | - | - | - | .0516 | .1268 | - | - |
| | 1.2115 | - | .0500 | - | .0487 | .1627 | .0979 | .3693 | .1430 | .1121 | - |
| | 1.2201 | .0413 | .0969 | - | .1526 | .3291 | .1089 | .1294 | - | - | - |
| | 1.3222 | .0391 | .0212 | - | .0366 | .0366 | - | .0465 | .0721 | - | .0231 |
| | 1.2129 | .0492 | .0556 | .0412 | .0436 | .1044 | .1440 | .2815 | .1406 | - | .0519 |
| | 1.2239 | .0538 | .0447 | .2804 | .0563 | .1266 | .0702 | .1752 | .0965 | .0224 | .0150 |
| 5. Primed PAA 1 mo. old | 1.2171 | .0570 | .0379 | .0314 | - | .0308 | - | - | - | .1307 | - |
| | 1.3067 | - | .0226 | - | - | .0680 | .0628 | - | - | - | .0668 |
| | 1.2485 | .0900 | - | - | .0392 | - | - | - | .0566 | - | .0428 |
| | 1.2877 | .0508 | .0308 | - | - | - | - | - | .0665 | - | .0338 |
| | 1.4220 | .0634 | .0377 | - | .0294 | .0431 | - | - | - | - | .1150 |
| | 1.2964 | .0522 | .0258 | .0063 | .0137 | .0284 | .0126 | - | .0246 | .0261 | .0517 |

APPENDIX IV (Continued)

II. 2024 T3 Clad Aluminum

| Surf. Prep. | Init. | 1 hr. | 4 hr. | 8 hr. | 1 day | 2 days | 3 days | 7 days | 14 days | 21 days | 36 days |
|--|--------|--------|-------|-------|-------|--------|--------|--------|---------|---------|---------|
| Wedge Crack Extension, inches | | | | | | | | | | | |
| 1. PAA (control) | 1.1643 | .0476 | .0374 | .0324 | - | .0368 | - | - | .0495 | - | .0521 |
| | 1.2648 | .0336 | - | .0463 | - | - | - | - | .0362 | - | .0565 |
| | 1.2125 | .0931 | - | - | .1099 | - | - | - | - | - | .0554 |
| | 1.2896 | .0305 | .0405 | - | .0314 | - | .0589 | - | - | - | .0538 |
| | 1.2347 | .0636 | .0391 | .0677 | - | - | - | - | - | - | .0706 |
| | 1.2332 | .0537 | .0234 | .0293 | .0283 | .0074 | .0118 | - | .0171 | - | .0577 |
| 2. PAA w/o OFPL deoxid. | 1.1122 | .0280 | .0272 | .0523 | .0683 | - | - | - | - | - | .0737 |
| | 1.1388 | .0492 | - | - | .0375 | .0910 | - | - | - | .1194 | - |
| | 1.1153 | .0831 | - | .0465 | .0473 | .0541 | - | - | - | - | .0483 |
| | 1.2075 | .0307 | .0432 | - | - | - | .0332 | - | - | - | .0887 |
| | 1.2895 | .0716 | .0683 | - | .0465 | - | - | - | - | - | .0518 |
| | 1.1727 | .0525 | .0277 | .0198 | .0399 | .0290 | .0078 | - | .0082 | .0239 | .0523 |
| 3. H ₂ SO ₄ deoxid./ PAA | 1.0968 | .1858 | - | - | - | - | - | - | .0413 | .0223 | .0567 |
| | 1.1017 | .1271 | - | - | - | .0298 | - | - | .0902 | - | .0545 |
| | 1.1541 | .1577 | .0342 | - | .0237 | - | - | - | - | .1111 | - |
| | 1.2504 | .0464 | .0320 | - | .0481 | - | - | - | .0402 | - | .0994 |
| | 1.2176 | .0711 | - | - | .0144 | .0060 | - | - | .0163 | .0267 | .0421 |
| | 1.1641 | .1176 | .0132 | - | - | - | - | - | - | - | - |
| 4. HNO ₃ (10%) | 1.2166 | 1.3715 | - | - | .0909 | - | - | - | - | - | - |
| | 1.2533 | 1.5571 | .0854 | - | - | - | - | - | - | - | .0629 |
| | 1.2244 | 1.6263 | .0658 | - | - | - | - | - | - | - | - |
| | 1.3389 | 1.4605 | - | .0657 | - | .0702 | - | - | - | - | - |
| | 1.1819 | 1.5427 | - | .1805 | .0307 | - | - | - | - | - | - |
| | 1.2430 | 1.5116 | .0302 | .0492 | .0243 | .0140 | - | - | - | - | .0126 |
| 5. Primed PAA 1 mo. old | 1.8953 | Fail | - | - | - | - | - | - | - | - | - |
| | 1.9935 | Fail | - | - | - | - | - | - | - | - | - |
| | 2.0291 | 2.523 | .594 | - | - | - | - | - | - | - | - |
| | 2.4216 | Fail | - | - | - | - | - | - | - | - | - |
| | 2.0915 | Fail | - | - | - | - | - | - | - | - | - |
| | 2.0862 | 2.523 | .594 | - | - | - | - | - | - | - | - |

III. 7075 T6 Bare Aluminum

| | | | | | | | | | | | |
|------------------|--------|-------|-------|-------|---|-------|-------|-------|-------|---|-------|
| 1. PAA (control) | 1.1684 | .1100 | - | .0519 | - | - | .0254 | - | - | - | .0947 |
| | 1.2716 | .0262 | - | - | - | .0350 | - | - | - | - | .0731 |
| | 1.2087 | .0747 | .0299 | .0268 | - | - | - | - | .0648 | - | .0702 |
| | 1.3335 | .0276 | .0421 | - | - | - | - | - | - | - | .0909 |
| | 1.2205 | .0434 | .0590 | - | - | .0070 | .0051 | .0416 | - | - | .0507 |
| | 1.2405 | .0564 | .0262 | .0157 | - | - | .0083 | .0083 | .0130 | - | .0759 |

APPENDIX IV (Continued)

| Surf. Prep. | Init. | 1 hr. | 4 hr. | 8 hr. | 1 day | 2 days | 3 days | 7 days | 14 days | 21 days | 30 days |
|--|--------|--------|-------|-------|-------|--------|--------|--------|---------|---------|---------|
| Wedge Crack Extension, Inches | | | | | | | | | | | |
| 2. PAA w/o deoxid. | 1.5312 | 3.6094 | Fail | | .0406 | - | - | .0473 | - | - | .0638 |
| | 1.6391 | Fail | | | - | .0409 | - | - | - | - | .0734 |
| | 1.7198 | Fail | | | - | - | - | - | .0727 | - | .0451 |
| | 1.6269 | 3.4844 | Fail | | .0343 | .0498 | - | - | - | - | .0578 |
| | 1.5938 | 3.5312 | Fail | | - | .0717 | - | - | - | .1020 | .0398 |
| 3. H ₂ SO ₄ deoxid./ PAA | 1.6222 | 3.5417 | | | - | - | - | .0095 | .0145 | .0204 | .0560 |
| | 1.2215 | .0240 | - | .0360 | .0406 | - | - | - | - | - | - |
| | 1.2598 | .0474 | - | - | - | .0409 | - | - | - | - | .0638 |
| | 1.2057 | .0510 | - | .0498 | - | - | - | - | .0727 | - | .0451 |
| | 1.2770 | .0473 | .0343 | .0717 | - | - | - | - | - | - | .0578 |
| 4. HNO ₃ (10%) | 1.4461 | .0511 | - | - | .0081 | .0082 | - | .0095 | .0145 | .0204 | .0398 |
| | 1.2820 | .0442 | .0068 | .0315 | .0081 | .0082 | - | .0095 | .0145 | .0204 | .0560 |
| | 1.2273 | .0236 | - | .0606 | .0577 | - | .1145 | .6743 | .1147 | - | .0320 |
| | 1.1929 | .4579 | - | .0454 | .1190 | .0704 | .0387 | - | - | - | - |
| | 1.2502 | .7671 | .0237 | - | .0389 | - | .0377 | - | - | - | .0702 |
| 5. Primed PAA 1 mo. old | 1.3267 | .0483 | .0051 | - | .5865 | .5865 | .0705 | .1183 | - | - | .0509 |
| | 1.4103 | .7369 | .0927 | .0663 | .0722 | - | .0705 | .1183 | .0409 | - | .0222 |
| | 1.2815 | .4068 | .0243 | .0345 | .0589 | .1314 | .0523 | .1585 | .0311 | - | .0351 |
| | 1.2219 | - | .0673 | - | .0512 | - | - | - | - | - | .0854 |
| | 1.2727 | - | - | - | - | .0360 | - | - | - | - | .0404 |
| | 1.1947 | .0906 | .0291 | - | - | .0502 | - | - | - | - | .0668 |
| | 1.2681 | .0681 | .0288 | .0465 | - | - | - | - | - | - | .0509 |
| | 1.2932 | - | - | - | - | .0316 | - | - | - | - | .0747 |
| | 1.2501 | .0317 | .0250 | .0093 | .0102 | .0236 | - | - | - | - | .0636 |
| | | | | | | | | | | | |

APPENDIX IV (Continued)

III. 2024 T3 Clad Aluminum

APPENDIX IV (Continued)

| Surf. Prep. | Init. | Wedge Crack Extension, Inches | | | | | | | | | |
|--|--------|-------------------------------|-------|-------|--------|--------|--------|-------|-------|-------|---------|
| | | 1 hr. | 4 hr. | 8 hr. | 24 hr. | 48 hr. | 72 hr. | 1 wk. | 2 wk. | 3 wk. | 30 days |
| 2. PA Deox./ PAA | 1.1768 | .0516 | - | - | - | - | .0885 | - | - | - | - |
| | 1.1488 | - | - | - | - | - | .0518 | - | - | - | - |
| | 1.2275 | .0560 | - | - | - | - | - | - | - | - | - |
| | 1.0793 | - | - | - | - | - | .0732 | - | - | - | .0824 |
| | 1.0967 | - | - | - | - | - | .0427 | - | - | - | .0165 |
| | 1.1462 | .0315 | - | - | - | - | - | - | - | - | - |
| 3. REV PA Deox./PAA | 1.1500 | - | .1596 | - | - | - | - | - | - | .0481 | .0287 |
| | 1.1749 | - | - | - | - | - | - | - | - | - | - |
| | 1.2003 | - | - | - | - | - | .0765 | - | - | - | - |
| | .9800 | - | - | - | - | - | - | - | - | - | - |
| | 1.1931 | - | .0319 | - | - | - | .0133 | - | - | .0096 | .0057 |
| | 1.1397 | - | - | - | - | - | - | - | - | - | - |
| 4. H ₂ SO ₄ Deoxid./ PAA | 1.1099 | - | .1596 | - | - | - | - | - | .0400 | - | - |
| | 1.2135 | - | - | - | - | - | - | - | - | - | - |
| | 1.2173 | - | - | - | - | - | - | - | - | - | - |
| | 1.1951 | - | - | - | - | - | - | - | - | - | - |
| | 1.1733 | .0754 | .0319 | - | - | - | - | - | .0080 | .0731 | - |
| | 1.1818 | .0151 | - | - | - | - | - | - | - | .0146 | - |
| 5. Scotch- brite/PAA | 1.3067 | - | - | - | - | - | - | .1347 | - | - | - |
| | 1.2131 | - | - | - | - | - | .0606 | - | - | - | - |
| | 1.2724 | - | - | - | - | - | - | - | - | - | - |
| | 1.2755 | - | - | - | - | - | - | - | .0412 | - | - |
| | 1.2506 | - | - | - | - | - | .0523 | - | - | - | - |
| | 1.2637 | - | - | - | - | - | .0226 | .0269 | .0083 | - | - |
| III. 7075 T6 Bare Aluminum | | | | | | | | | | | |
| 1. PAA (control) | 1.4439 | - | - | - | - | - | - | - | - | - | - |
| | 1.1432 | .0328 | - | - | - | - | .0605 | - | - | - | - |
| | 1.2497 | - | - | - | - | - | - | - | - | .0715 | - |
| | 1.3627 | - | - | - | - | - | - | - | - | - | - |
| | 1.3367 | - | - | - | - | - | .0927 | - | - | - | - |
| | 1.3072 | .0066 | - | - | - | - | .0306 | - | - | .0179 | - |
| 2. PA Deox./ PAA | 1.2998 | - | - | - | - | - | - | - | - | - | - |
| | 1.2243 | - | - | - | - | - | - | - | - | - | - |
| | 1.2274 | - | - | - | - | - | - | - | - | .0572 | - |
| | 1.2997 | - | - | - | - | - | - | - | - | - | - |
| | 1.2667 | - | - | - | - | - | - | - | - | - | - |
| | 1.2636 | - | - | - | - | - | - | - | - | .0114 | - |

APPENDIX IV (Continued)

| Surf. Prep. | Init. | 1 hr. | 4 hr. | 8 hr. | 24 hr. | 48 hr. | 72 hr. | 1 wk. | 2 wk. | 3 wk. | 30 days |
|-----------------------------------|--------|-------|-------|-------|--------|--------|--------|-------|-------|-------|---------|
| Wedge Crack Extension, inches | | | | | | | | | | | |
| 3. REV PA | 1.2505 | - | - | - | - | - | .0998 | - | - | - | - |
| Deox./ | 1.3116 | - | - | - | - | - | - | .0341 | - | - | - |
| PAA | 1.2396 | .1118 | - | - | - | - | - | - | - | - | - |
| | 1.2282 | - | - | - | - | - | - | - | - | - | - |
| | 1.4746 | - | - | - | - | - | .0200 | .0068 | - | - | - |
| | 1.3009 | .0224 | - | - | - | - | - | - | - | - | - |
| 4. H ₂ SO ₄ | 1.3363 | - | - | .0729 | - | - | - | - | - | - | - |
| Deox./ | 1.2447 | - | - | - | - | - | - | - | - | - | - |
| PAA | 1.1911 | - | - | - | - | - | .0479 | - | - | - | - |
| | 1.3407 | - | - | - | - | - | - | - | - | .0379 | - |
| | 1.2651 | - | - | - | - | - | .0661 | - | - | - | - |
| | 1.2756 | - | - | .0146 | - | - | .0028 | - | - | .0076 | - |
| 5. Scotch- | 1.2471 | - | - | - | - | - | - | - | - | .0446 | - |
| brite/PAA | 1.2674 | - | - | - | - | - | - | - | - | .1085 | - |
| | 1.2730 | - | .1531 | - | - | - | - | - | - | - | - |
| | 1.3075 | - | - | - | - | - | - | - | - | - | - |
| | 1.3554 | - | - | - | - | - | - | - | - | - | - |
| | 1.2901 | - | .0306 | - | - | - | - | - | - | .0383 | - |

APPENDIX IV (Continued)

INDIVIDUAL WEDGED CRACK EXTENSION TEST RESULTS FOR SET D

| I. 2024 T3 Bare Aluminum | | | | | | | | | | | |
|-------------------------------|--------|--------|-------|-------|--------|--------|--------|-------|-------|-------|---------|
| Surf. Prep. | Init. | 1 hr. | 4 hr. | 8 hr. | 24 hr. | 48 hr. | 72 hr. | 1 wk. | 2 wk. | 3 wk. | 30 days |
| Wedge Crack Extension, Inches | | | | | | | | | | | |
| 1. PAA (control) | 1.3795 | - | - | - | .0850 | - | - | - | - | - | .0415 |
| | 1.3722 | .0401 | - | - | - | - | - | .0632 | - | - | - |
| | 1.3467 | - | - | - | - | - | .0441 | - | - | - | - |
| | 1.2868 | - | - | - | - | - | - | - | .0434 | - | - |
| | 1.5094 | - | - | - | - | - | - | - | - | - | - |
| | 1.3789 | - | - | - | - | - | - | - | - | - | - |
| 2. P2 | 1.4161 | 1.7024 | .2818 | - | - | - | - | - | - | - | - |
| | 1.5369 | 1.9079 | .1362 | - | - | - | - | - | - | - | - |
| | 2.3458 | 1.2643 | .2559 | - | - | - | .1274 | - | - | - | - |
| | 2.4295 | 1.4283 | Fail | - | - | - | - | - | - | - | - |
| | 2.3308 | Fail | - | - | - | - | - | - | - | - | - |
| | 2.0118 | - | - | - | - | - | - | - | - | - | - |
| II. 2024 T3 Clad Aluminum | | | | | | | | | | | |
| 1. PAA (control) | 1.2192 | - | - | - | .0333 | - | - | .0234 | - | - | - |
| | 1.1615 | - | - | - | - | - | - | .0261 | - | - | - |
| | 1.0734 | - | - | - | - | - | .0343 | - | - | - | .0549 |
| | 1.1699 | - | - | - | - | - | - | - | - | - | - |
| | 1.1514 | - | - | - | - | - | - | - | - | - | .0558 |
| | 1.1551 | - | - | - | - | - | - | - | - | - | - |
| 2. P2 | 1.1102 | .0597 | - | - | - | - | - | - | - | - | - |
| | 1.0857 | .3657 | - | - | - | - | - | - | .0801 | - | - |
| | 1.0142 | .4681 | - | - | - | - | - | - | - | - | - |
| | 1.0159 | .7899 | - | - | - | - | - | - | - | - | - |
| | 1.0703 | - | - | - | - | - | - | - | - | - | - |
| | 1.0593 | .4209 | - | - | - | - | - | - | - | - | - |
| III. 7075 T6 Bare Aluminum | | | | | | | | | | | |
| 1. PAA (control) | 1.2994 | - | - | - | .0911 | - | - | - | .0596 | - | - |
| | 1.2128 | - | - | - | - | - | - | - | - | - | .0718 |
| | 1.2015 | - | - | - | - | - | - | - | - | - | - |
| | 1.3310 | - | - | - | - | - | - | - | - | - | - |
| | 1.3442 | - | - | - | - | - | - | - | - | - | - |
| | 1.2778 | - | - | - | - | - | - | - | - | - | - |

APPENDIX IV (Continued)

| Surf. Prep. | Init. | 1 hr. | 4 hr. | 8 hr. | 24 hr. | 48 hr. | 72 hr. | 1 wk. | 2 wk. | 3 wk. | 30 days |
|------------------------------------|--------|--------|-------|-------|--------|--------|--------|-------|-------|-------|---------|
| Wedged Crack Extension, Inches | | | | | | | | | | | |
| III. 7075 T6 Bare Aluminum (cont.) | | | | | | | | | | | |
| 2. P2 | 1.3511 | 2.1582 | .0945 | - | - | - | - | - | - | - | - |
| | 1.3948 | 2.7982 | .0937 | - | - | - | - | - | - | - | - |
| | 1.3622 | 3.7236 | Fail | - | - | - | - | - | - | - | - |
| | 1.4622 | 1.3489 | - | - | - | - | - | .6149 | .1052 | - | - |
| | 1.3263 | .3193 | - | - | - | .9060 | .3744 | .1099 | - | - | - |
| | 1.3793 | 2.0696 | | | | | | | | | |

-- No Change

All samples were primed with BR 127 to a thickness of 0.0001-0.0002 inch.

All samples were bonded with EA9628 adhesive.

The crack lengths are not cumulative.

APPENDIX IV (Continued)
INDIVIDUAL WEDGED CRACK EXTENSION TEST RESULTS FOR SET E

| Surf. Prep. | Init. | 1 hr. | 4 hr. | 8 hr. | 24 hr. | 48 hr. | 72 hr. | 1 wk. | 2 wk. | 3 wk. | 30 days |
|----------------------------|--------|--------|-------|-------|--------|--------|--------|-------|-------|-------|---------|
| I. 2024 T3 Bare Aluminum | | | | | | | | | | | |
| 1. PAA (control) | 1.4723 | .0249 | - | - | - | - | - | .0456 | - | - | - |
| | 1.3759 | .0389 | - | - | - | - | .0539 | - | - | - | - |
| | 1.4952 | - | - | - | - | - | - | - | - | - | - |
| | 1.3896 | - | - | - | - | - | - | .0192 | - | - | - |
| | 1.4197 | - | - | - | - | - | - | - | - | - | - |
| | 1.4305 | - | - | - | - | - | - | - | - | - | - |
| 2. P2 | 1.3618 | 1.3281 | - | - | - | - | - | - | - | - | - |
| | 1.7403 | 1.7373 | .1152 | - | - | - | - | - | - | - | - |
| | 2.0454 | 1.5806 | - | .1127 | - | - | - | - | - | - | - |
| | 1.9075 | 1.5632 | - | - | - | - | - | .0384 | - | - | - |
| | 1.2450 | .1287 | - | - | .0580 | - | - | - | .0463 | - | - |
| | 1.6660 | 1.2676 | - | - | - | - | - | - | - | - | - |
| II. 2024 T3 Clad Aluminum | | | | | | | | | | | |
| 1. PAA (control) | 1.1366 | - | - | - | - | - | - | - | - | - | - |
| | 1.1292 | - | - | - | - | - | - | - | - | - | .0371 |
| | 1.2418 | - | - | - | - | - | - | - | - | - | .0287 |
| | 1.2373 | - | - | - | - | - | - | - | - | - | - |
| | 1.2559 | - | .0194 | - | - | - | - | - | - | - | - |
| | 1.2002 | - | - | - | - | - | - | - | - | - | - |
| 2. P2 | 1.3714 | 1.8430 | - | .1625 | - | - | - | - | - | - | - |
| | 1.3410 | 2.1890 | - | .0489 | - | - | - | - | - | - | - |
| | 1.2738 | 1.5472 | - | - | - | - | - | - | - | - | - |
| | 1.0115 | 1.6502 | .0432 | - | - | - | - | .0547 | - | - | - |
| | 1.1721 | .4950 | - | - | - | - | - | - | - | .0795 | - |
| | 1.2340 | 1.5449 | - | - | - | - | - | - | - | - | - |
| III. 7075 T6 Bare Aluminum | | | | | | | | | | | |
| 1. PAA (control) | 1.3006 | .0344 | - | - | - | - | - | - | - | - | - |
| | 1.3009 | - | - | - | - | - | .0632 | - | - | - | - |
| | 1.3925 | - | - | - | - | - | - | - | - | - | - |
| | 1.3651 | .0253 | - | - | - | - | - | - | - | - | - |
| | 1.3976 | - | - | - | - | - | - | - | - | - | - |
| | 1.3513 | - | - | - | - | - | - | - | - | - | - |

APPENDIX IV (Concluded)

| Surf. Prep. | Init. | 1 hr. | 4 hr. | 8 hr. | 24 hr. | 48 hr. | 72 hr. | 1 wk. | 2 wk. | 3 wk. | 30 days |
|------------------------------------|---------------|-------|-------|-------|--------|--------|--------|-------|-------|-------|---------|
| III. 7075 T6 Bare Aluminum (cont.) | | | | | | | | | | | |
| 2. P2 | 1.5038 | - | - | - | - | - | - | .1512 | .3294 | - | - |
| | 1.3640 | .7554 | - | - | .0511 | - | - | .1117 | - | - | .0899 |
| | 1.3815 | .8972 | - | - | - | - | - | - | - | - | - |
| | 1.5454 | - | - | - | .1742 | - | - | .3132 | .0795 | - | - |
| | 1.2784 | - | - | - | .0309 | - | - | .7879 | - | - | - |
| | <u>1.4146</u> | | | | | | | | | | |

APPENDIX V

FLOATING ROLLER PEEL TEST RESULTS

Listed below are the individual results for the floating roller peel tests for sets E1 through E6. The samples are listed in groups of five; each group of five was taken from the same panel. This permits ready comparison of the failure modes obtained for each test condition from any one panel. As noted below, six panels consistently had very low peel strength and high metal to primer failure regardless of the test conditions.

| Test Condition | No. | E1-PAA (1) | | | E2-PAD/PAA | | | E3-SAD/PAA | | | E4-PAA (no deox.) | | | E5-Sc-MEK/PAA | | | E6-P2 (no anod.) | | |
|----------------|-----|-------------------------------|---------------------|----------|-------------------------------|-----------------|----------|-------------------------------|-----------------|----------|-------------------------------|-----------------|-------|-------------------------------|-----------------|-------------|-------------------------------|-----------------|---|
| | | Peel Strength (lbs/in. width) | Failure Mode, % (2) | | Peel Strength (lbs/in. width) | Failure Mode, % | | Peel Strength (lbs/in. width) | Failure Mode, % | | Peel Strength (lbs/in. width) | Failure Mode, % | | Peel Strength (lbs/in. width) | Failure Mode, % | | Peel Strength (lbs/in. width) | Failure Mode, % | |
| | | | MP | PA | | A | V | | MP | PA | | A | V | | MP | PA | | A | V |
| -65°F | 13 | 30(1) | 6/5 | 0 30 5 | 19(3) | 6/0 | 5 30 5 | 56 | 25 | 30 35 10 | 59 | 5 35 50 10 | 65 | 5 40 40 15 | 58 | 0 45 25 30 | | | |
| 72°F | 16 | 17 | 9/0 | 5 5 0 | 16 | 9/0 | 5 5 0 | 88 | 0 | 50 30 20 | 79 | 5 0 85 10 | 85 | 0 30 55 15 | 81 | 0 30 50 20 | | | |
| 180°F | 19 | 20 | 9/5 | 0 0 5 | 32 | 9/5 | 5 0 0 | 96 | 5 | 10 60 25 | 91 | 0 15 75 10 | 95 | 0 10 70 20 | 89 | 5 0 70 25 | | | |
| -65Exp | 22 | 12 | 7/5 | 0 5 20 | 10 | 8/0 | 0 10 10 | 44 | 0 | 60 15 25 | 55 | 0 0 80 20 | 57 | 0 55 35 10 | 55 | 0 35 35 30 | | | |
| 180Exp | 25 | 45 | 9/0 | 0 0 10 | 24 | 9/0 | 0 0 10 | 84 | 0 | 0 70 30 | 85 | 0 0 90 10 | 92 | 0 0 80 20 | 85 | 10 0 65 25 | | | |
| -65°F | 14 | 58 | 5 | 25 65 5 | 33(3) | 4/5 | 10 30 15 | 59 | 0 | 60 30 10 | 59 | 10 20 60 10 | 8(3) | 5 5 25 5 | 52 | 15 45 20 20 | | | |
| 72°F | 17 | 69 | 5 | 10 85 0 | 14 | 7/0 | 0 10 20 | 84 | 0 | 30 60 10 | 82 | 0 20 65 15 | 4 | 80 0 0 20 | 63 | 35 10 35 20 | | | |
| 180°F | 20 | 78 | 10 | 20 60 10 | 9 | 8/5 | 5 0 10 | 84 | 0 | 0 90 10 | 90 | 0 0 85 15 | 6 | 85 0 0 15 | 72 | 70 0 20 10 | | | |
| -65Exp | 23 | 56 | 0 | 45 45 10 | 5 | 9/0 | 0 0 10 | 56 | 0 | 20 70 10 | 54 | 0 0 80 20 | 5 | 90 0 0 10 | 45 | 0 35 35 30 | | | |
| 180Exp | 26 | 74 | 0 | 0 95 5 | 18 | 9/0 | 0 0 10 | 78 | 0 | 0 90 10 | 94 | 0 0 85 15 | 6 | 65 0 0 5 | 75 | 60 0 10 30 | | | |
| -65°F | 15 | 60 | 5 | 40 45 10 | 55 | 2/5 | 15 55 5 | 62 | 5 | 10 75 10 | 4(3) | 90 0 0 10 | 50(3) | 25 25 30 20 | 47 | 20 50 15 15 | | | |
| 72°F | 18 | 77 | 5 | 30 35 30 | 78 | 10 | 15 60 15 | 76 | 0 | 5 90 5 | 4 | 90 0 0 10 | 43 | 40 10 10 40 | 63 | 50 5 30 15 | | | |
| 180°F | 21 | 81 | 0 | 20 60 20 | 81 | 15 | 0 70 15 | 87 | 0 | 20 70 10 | 3 | 90 10 0 0 | 37 | 50 0 5 45 | 88 | 20 5 70 5 | | | |
| -65Exp | 24 | 56 | 0 | 40 45 15 | 56 | 0 | 10 90 10 | 57 | 0 | 15 75 10 | 6 | 90 0 0 10 | 11 | 15 25 0 60 | 53 | 0 35 35 30 | | | |
| 180Exp | 27 | 85 | 0 | 0 80 20 | 78 | 4/0 | 0 50 10 | 88 | 0 | 0 85 15 | 4 | 90 0 0 10 | 65 | 60 0 5 35 | 82 | 60 0 10 30 | | | |

(1) PAA - Phosphoric Acid Anodizing; SAD - Sulfuric Acid Deoxidizing; P2 - P2 Etch; PAD - Phosphoric Acid Deoxidizing; Sc-MEK - Scotchbrite Pad Abrasion with MEK Wipe, no other deoxidizing.

(2) Failures Modes: MP - Metal to primer; PA - Primer to adhesive; A - Cohesive Adhesive Failure; V - Voids.

(3) Six groups of results were not used in determining the averages as reported in Table 3 due to the high percentage of metal to primer failure for all of the samples in those groups.

APPENDIX VI

DOUBLE LAP SHEAR TEST RESULTS

Listed below are the individual results for Sets E1 through E6 for the double lap shear tests. In several cases, there was a wide range of results. The lower results usually could be attributed to high bondline void content. If these values were not used in the averages, the results were consistently in line with the other samples tested under the same conditions.

| Test Condition | No. | E1-PAA(1) | | | E2-PAD/PAA | | | E3-SAD/PAA | | | E4-PAA (no deot.) | | | E5-Sc-MEX/PAA | | | E6-P2 (no anod.) | | |
|----------------|-----|----------------------|-----------------|----|------------|----------|----|----------------------|-----------------|----|-------------------|----------|----|----------------------|-----------------|----|------------------|----------|----|
| | | Shear Strength (psi) | Failure Mode, % | MP | PA | A | V | Shear Strength (psi) | Failure Mode, % | MP | PA | A | V | Shear Strength (psi) | Failure Mode, % | MP | PA | A | V |
| -65°F | 1 | 8138 | 10 20 70 | 0 | 8036 | 5 10 85 | 0 | 7627 | 0 35 65 | 0 | 8116 | 10 0 90 | 0 | 8517 | 15 0 85 | 0 | 7989 | 0 40 60 | 0 |
| | 2 | 7596 | 15 0 85 | 0 | 8216 | 20 0 80 | 0 | 6241 | 10 0 75 | 15 | 6414 | 20 15 35 | 5 | 7862 | 35 15 50 | 0 | 5322 | 25 0 75 | 0 |
| | 3 | 7881 | 5 65 30 | 0 | 7582 | 10 0 80 | 10 | 7056 | 10 0 45 | 40 | 7638 | 10 50 40 | 0 | 8515 | 0 0 100 | 0 | 7909 | 20 0 30 | 50 |
| 72°F | 4 | 7872 | 10 25 65 | 0 | 7945 | 5 10 85 | 0 | 6975 | 10 0 45 | 5 | 7390 | 10 0 90 | 0 | 8298 | 15 0 85 | 0 | 7071 | 20 0 80 | 0 |
| | 5 | 7167 | 15 0 85 | 0 | 6816 | 10 0 80 | 10 | 5944 | 10 0 85 | 5 | 5077 | 10 0 90 | 0 | 7059 | 15 0 85 | 0 | 4499 | 10 10 75 | 5 |
| | 6 | 6979 | 10 0 90 | 0 | 6837 | 0 0 100 | 0 | 5743 | 0 0 100 | 0 | 6259 | 0 0 100 | 0 | 6900 | 0 0 100 | 0 | 6609 | 5 0 95 | 0 |
| 180°F | 7 | 7122 | 10 0 90 | 0 | 7020 | 0 0 100 | 0 | 6164 | 10 0 80 | 10 | 5913 | 10 0 80 | 10 | 6722 | 0 0 100 | 0 | 5528 | 10 10 80 | 0 |
| | 8 | 6268 | 5 0 90 | 5 | 6891 | 20 10 60 | 10 | 5950 | 10 0 65 | 25 | 5748 | 0 0 100 | 0 | 6894 | 20 10 60 | 10 | 5545 | 0 0 95 | 5 |
| | 9 | 6093 | 10 0 90 | 0 | 6135 | 25 0 75 | 0 | 5339 | 45 0 55 | 0 | 5493 | 20 0 80 | 0 | 5608 | 15 0 85 | 0 | 5892 | 30 0 65 | 5 |
| 180°F Exp. | 10 | 3892 | 10 0 90 | 0 | 5856 | 30 0 70 | 0 | 4909 | 25 0 60 | 15 | 4352 | 15 0 80 | 5 | 5807 | 0 0 95 | 5 | 4357 | 30 0 70 | 0 |
| | 11 | 4366 | 30 15 55 | 0 | 5737 | 15 0 85 | 0 | 5612 | 10 0 90 | 0 | 5036 | 35 0 40 | 25 | 6827 | 30 0 70 | 0 | 5566 | 50 0 50 | 0 |
| | 12 | 4303 | 15 0 85 | 0 | 5910 | 5 0 90 | 0 | 4953 | 30 0 50 | 20 | 4960 | 0 10 85 | 5 | 6081 | 30 0 70 | 0 | 5272 | 25 0 40 | 30 |
| | 13 | 6057 | 0 10 90 | 0 | 4000 | 50 0 45 | 5 | 3569 | 25 0 55 | 20 | 3703 | 15 0 80 | 5 | 6081 | 0 0 100 | 0 | 5272 | 60 0 40 | 0 |
| | 14 | 3892 | 50 0 50 | 0 | 3448 | 20 0 80 | 0 | 3385 | 10 0 90 | 0 | 2238 | 50 0 0 | 50 | 4038 | 5 0 90 | 0 | 2607 | 40 50 10 | 0 |
| | 15 | 4366 | 10 0 100 | 0 | 3964 | 0 0 85 | 15 | 3385 | 50 0 25 | 25 | 3703 | 0 0 100 | 0 | 5865 | 20 0 80 | 0 | 4205 | 10 0 30 | 60 |
| | 16 | 4303 | 10 0 90 | 0 | 3804 | 50 0 25 | 25 | 4366 | 0 0 100 | 0 | 2528 | 5 0 95 | 0 | 4163 | 0 0 100 | 0 | 2957 | 20 0 80 | 0 |
| | 17 | 4187 | 75 0 25 | 0 | 3964 | 0 0 100 | 0 | 4366 | 0 0 100 | 0 | 2528 | 90 0 0 | 10 | 4163 | 40 0 60 | 0 | 2957 | 50 0 50 | 0 |
| | 18 | 4187 | 0 0 90 | 10 | 3804 | 0 0 90 | 10 | 3774 | 30 0 60 | 10 | 2823 | 40 0 60 | 0 | 4689 | 0 0 95 | 5 | 3257 | 25 0 25 | 50 |

(1) PAA - Phosphoric Acid Anodizing; SAD - Sulfuric Acid Deoxidizing; P2 - P2 etch; PAD - Phosphoric Acid Deoxidizing; Sc-MEX - Scotchbrite Pad Abrasion with MEX wipe.

(2) Failure Modes: MP - Metal to primer; PA - Primer to adhesive; A - Cohesive adhesive failure; V - Voids.